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PROCEEDINGS

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DP3

An Interrupted Segmented Flow Stream Microwave Solid Sample Decomposition System for ICP-AES

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Microwave aided digestion offers significant speed advantages over conventional atmospheric or bomb digestion techniques for certain sample types (1-4). It is our hope to develop a flowing stream microwave system suitable to the digestion of solid samples. It is our expectation that such a system would provide many of the throughput advantages enjoyed by segmented and flow injection systems. In addition, automated handling of liquids should minimize the fume hazards which can result when using the more powerful reagents required for some difficult digestions.

Sample, as a 0.2 g slurry in 25 ml is aspirated into tubing where it is mixed at a "T" junction with 25 ml of 0.2 M reagent grade nitric acid. During this process Valves 1 and 2 are open. A conventional "kitchen" type microwave oven has been modified by the addition of two 3/8 in holes for the entrance and exit of the tubing and the addition of an exhaust fan to aid in the removal of hot air. While this oven has operated properly, we encourage researchers to buy industrial calibre microwave ovens for several reasons, (1) they can tolerate higher internal temperatures and (2) they allow considerably more flexibility in their heating cycles.

Initially, Valves 1 and 2 are open. The slurry/acid solution is pumped into the microwave and Valves 1 and 2 are closed. The solution is then subjected to the microwave radiation. At the completion of the exposure, the system is allowed to cool approximately 1 minute. Valve 2 is then opened to relieve pressure. Any effluent is collected in the collection vessel. Then Valve 1 is opened and the tubes are removed from the slurry solution and the acid reservoir. The sample plug is followed by an air bubble. The system can be cleaned by an acid plug or rinsed with a water plug or acid plug.

The demonstration problem undertaken is one common to one of our undergraduate laboratories, the digestion of Cu Ore using nitric acid. For comparison purposes, we have carried out experiments using the apparatus illustrated in Figure 1 and using a traditional atmospheric hot plate digestion procedure. The sample was in a 325 mesh (44 micron) particle size format. The sample was used in exactly the same format with the same reagents in both experiments.

The results are presented succinctly in Figure 2. Compared with conventional atmospheric digestion conditions, the stream microwave system is an order of magnitude faster. Both the temperature and the pressure are measurably elevated over those available at atmospheric pressure. These experiments were designed to test the effectiveness of the energy coupling into tubing of the dimensions employed. They were obviously a success in this regard. We hope to develop a continuous flow system for rapid continuous digestion of solid samples.

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Microwave Solid Stream Digestion Apparatus
First Experimental Configuration

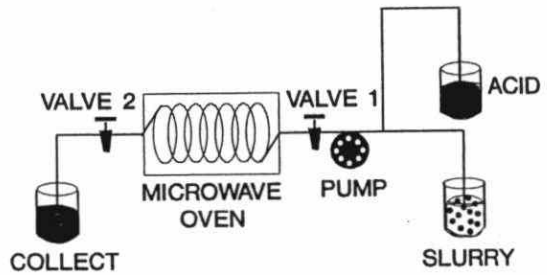

Figure 1

Digestion Times
0.2 g of Cu Ore

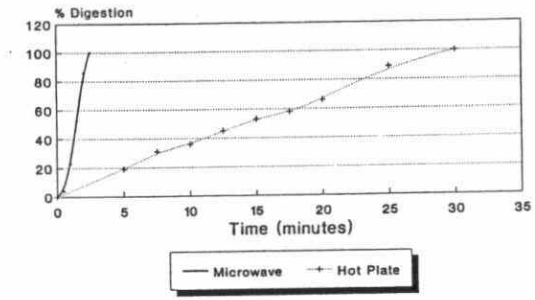

Figure 2